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(पहला पुनरीक्षण)

Indian Standard

GUIDELINES FOR ASH SOFTENING STUDIES ON SOLID REDUCTANTS FOR DIRECT REDUCTION

(First Revision)

ICS 77.020

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

August 1996

Price Group 2

Sponge Iron and Smelting Reduction Sectional Committee, MTD 30

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Sponge Iron and Smelting Reduction Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard was first published in 1986. While reviewing the standard in the light of the experience gained during these years, the Committee had decided that the standard may be revised. In this revision, various clauses have been aligned with the recent practices being followed. The following modifications have been made in the revised standard:

a) Definition of ash characteristics, temperatures have been modified.

b) The values for maximum acceptable difference (repeatability) have been reduced.

Suitability of a solid reductant in making sponge iron/direct reduced iron (DRI) depends, amongst others, on the ash softening characteristics of the same. As a result, increasing attention is being paid to assess this property to attain higher kiln availability and longer compaign life.

Ash softening characteristics are determined under both oxidizing and reducing conditions and in this standard, the typical acceptable values have been indicated.

Ash softening characteristcs of solid reductant are useful in selecting the optimum kiln operating temperature. However, it must be remembered that within the kiln, the coal ash gets intimately mixed with many other chemical species, forming a variety of possible cutectic mixtures whose melting points may be significantly lower than that of the pure ash. The ash softening data, therefore, should be used for initial evaluation only.

In reporting the results of test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

AMENDMENT NO. 1 NOVEMBER 2007
TO
IS 11795 : 1996 GUIDELINES FOR ASH SOFTENING STUDIES ON SOLID REDUCTANTS FOR DIRECT REDUCTION

(First Revision)

(*Page* 1, *clause* **1.1**, *line* 3) — Substitute 'reductants' *for* 'reductant'.

(*Page* 1, *clause* **2.1**, *Heading*) — Substitute '**Initial Deformation Temperature**' *for* '**Ash Softening Temperature**'.

(*Page* 1, *clause* **2.2**, *Heading*) — Substitute '**Hemispherical Temperature**' *for* '**Ash Fusion Temperature**'.

(*Page* 2, *clause* **5.1**, *line* 2) — Substitute 'of' *for* 'or'.

(*Page* 2, *clause* **5.1**, *line* 7) — Substitute 'a' *for* 'an'.

(*Page* 2, *clause* **7.2**, *line* 4) — Insert 'Full stop' after 'bubbles'. Delete 'and' and substitute 'In' *for* in'.

(*Page* 2, *clause* **7.3**, *line* 1) — Substitute 'observations' *for* 'observation'.

(*Page* 2, *clause* **8.1**, *line* 2) — Substitute 'results' *for* 'result'.

(*Page* 3, *clause* **8.2**, *line* 4) — Substitute 'preparation' *for* 'oreparation'.

(*Page* 3, *clause* **8.2**, *line* 5) — Substitute 'amount' *for* 'amounts'.

(MTD 30)

Reprography Unit, BIS, New Delhi, India

Indian Standard

GUIDELINES FOR ASH SOFTENING STUDIES ON SOLID REDUCTANTS FOR DIRECT REDUCTION

(First Revision)

1 SCOPE

1.1 This standard covers the guidelines for determination of softening characteristic of ash using a heating microscope for all solid reductant used in direct reduction in making sponge iron/direct reduced iron (DRI).

2 TERMINOLOGY

For the purpose of this standard, the following definitions shall apply.

2.1 Ash Softening Temperature

It is the temperature at which the first sign of rounding of the edges of the ash cube or cylinder occur. This may also be called Initial Deformation Temperature and termed as IDT (Fig. 1A and Fig. 1B).

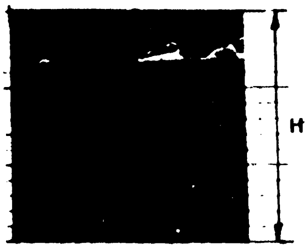

FIG. 1A ORIGINAL SHAPE OF THE SAMPLE

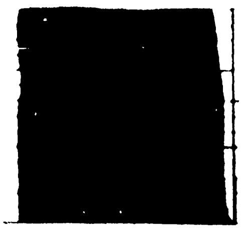

FIG. 1B SHAPE OF THE SAMPLE AT SOFTENING TEMPERATURE

2.2 Ash Fusion Temperature

It is the temperature at which the ash fuses completely into a hemispherical blob, for example when the height of the sample becomes equal to half the base diameter, as observed visually. This may also be called Ash Melting Temperature and termed as AFT (Fig. 2).

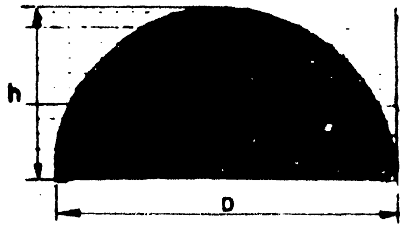

FIG. 2 SHAPE OF THE SAMPLE AT ASH FUSION / HEMISPHERICAL TEMPERATURE – $h = \frac{1}{2} D$

2.3 Ash Flow Point

It is the temperature at which the ash sample spreads out in a flat layer, the height of which is approximately equal to one-third of that of the test piece at the ash fusion temperature and termed as FFT (Fig. 3)

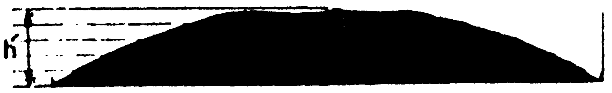

FIG. 3 SHAPE OF THE SAMPLE AT THE ASH FLOW POINT
$h' = \frac{1}{3} H$

3 PRINCIPLE

A test piece made from the ash is heated under specified conditions and is continuously observed. The temperature at which characteristic changes of shape occur, are recorded. The ash characteristic temperatures are given at **2**.

4 TEST CONDITIONS

4.1 Shape of Test Piece

The test piece shall have sharp edges to facilitate observation and shall not bend during the determination. The mass of the test piece shall be such that it ensures equalization of temperature within the test body. Hence, too large dimensions shall be avoided.

The following shapes and dimensions are acceptable.

a) Cube with each side as 2 mm or 3 mm, and

b) Cylinder with dia 2 mm or 3 mm and height equal to its diameter.

The coal ash can be moistened with a few drops of clear filtered 10 percent solution of the dextrin containing 0.1 percent salicylic acid as a preservative and the sample prepared manually.

4.2 Test Atmosphere

4.2.1 *Mild Reducing Atmosphere*

An atmosphere in which the volume ratio of reducing gases to oxidizing gases falls within limits 4 : 1 and 1 : 4 respectively. For this purpose, carbon dioxide and water vapour are considered as oxidizing gases and hydrogen and carbon monoxide are considered as reducing gases.

4.2.2 *Oxidizing Atmosphere*

An atmosphere in which the volume ratio of reducing gases to oxidizing gases is less than 1 : 4. The oxidizing atmosphere is obtained with air or carbon dioxide but the rate of flow is not critical.

5 APPARATUS

5.1 The heating microscope permits the observation and photographic recording or various phases of the characteristic changes of physical state of ash. Samples of substances to be investigated are microscopically observed during heating in an electric furnace and their images can be projected on a 24 mm × 36 mm ground glass screen. In an detachable camera, the entire process of heating the sample up to the fusion point or melting point may be recorded by taking a series of photographs.

5.2 Furnace

The furnace is electrically heated and is capable of:

a) reaching the maximum temperature at which the properties of the ash are to be determined (a temperature of 1 600°C or higher, if required);

b) providing an adequate zone of uniform temperature to heat the test piece;

c) providing means of heating the test piece at a uniform rate, the maximum being 10°C/min;

d) maintaining around the test piece, an atmosphere of composition within the desired limits; and

e) providing means of observing the change of shape of test piece during heating.

5.3 Pyrometer

The temperature shall be measured by means of platinum rhodium thermocouple. The calibration of the pyrometer shall be carried out from time to time.

5.4 Mould for Preparing the Test Piece

Sample shall be mounted by hand pressing for the preparation of cubical sample up to 3 mm in height and cylindrical samples up to 3 mm in diameter.

5.5 Support for the Test Piece

The support shall be made of such a material that it shall neither get distorted, nor shall it react with the constituents. It shall also not absorb ash during the determination. Generally, the sample support may be made of steatite, sipolax or aluminium oxide (Al_2O_3).

5.6 Optical Instrument

An instument which enables the profile of test piece to be observed throughtout the determination. The relative dimensions may be conveniently assessed by the use of a graticule.

6 PREPARATION OF TEST PIECE

The ash sample is prepared by igniting coal samples in a well ventilated muffle furnace maintained at 800°C. It should be ensured that the incineration of the sample is complete. The ash sample is ground in an agate mortar until the maximum particle size is 100 percent less than 0.076 mm.

7 PROCEDURE

7.1 The test piece is transferred on its support to the furnace and the furnace is preheated to a temperature of 815°C. The temperature of the furance is raised at the rate of about 10°C/min. At 815°C, the composition and flow rate of the atmosphere is adjusted according to the test conditions. The temperatures are recorded at which the characteristic changes of shape occur. The slower rate of heating is preferable for larger test pieces. However, for smaller test pieces, a rate of temperature rise up to 10°C/min may be satisfactory.

7.2 With some ashes, difficulties may be encountered owing to such effects as blistering, distortion, shrinkage, swelling, non-wetting of the support and bursting of internal gas bubbles and in such cases it is desirable to record these phenomena and possibly repeat the experiment using a different type of support.

7.3 The observation are recorded at intervals of 20°C approximately and after the temperature of 1 000°C is reached, the following three defined temperatures are noted specifically:

a) Ash softening temperature,

b) Ash fusion temperature, and

c) Ash flow point.

8 PRECISION OF TEST METHOD

8.1 The maximum acceptable differences among the result shall be as given in Table 1.

Table 1 Maximum Acceptable Differences Between the Results

(*Clause* 8.1)

Sl No.	Softening Characteristics	Maximum Acceptable Differences (Repeatability)
i)	Ash softening temperature	25 °C
ii)	Ash fusion temperature	25 °C
iii)	Ash flow point	40 °C

8.2 Repeatability

The results of duplicate determinations carried out at different times in the same laboratory by the same operator with the same apparatus on the same oreparation of ash, shall not differ by more than the above amounts.

9 TEST RESULTS

9.1 The test report shall state:

i)	Ash softening temperature	Each rounded off to the nearest 10 °C
ii)	Ash Fusion temperture	
iii)	Ash flow point	

iv) Size and shape of the test piece,

v) Composition of the atmosphere, and

vi) Nature of the support.

It is also desirable to report any phenomena which could have influenced the results.

9.2 Typical acceptable values for solid reductants used in direct reduction are as follows:

Softening Characteristics	*Oxidizing Conditions (Indicative)*	*Mild Reducing Conditions (Indicative)*
Ash softening temperature	1 200 °C, *Min*	1 150 °C, *Min*
Ash fusion temperature	1 300 °C, *Min*	1 225 °C, *Min*
Ash flow point	1 400 °C, *Min*	1 275 °C, *Min*

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards : Monthly Additions'.

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